EUR 4807 e

COMMISSION OF THE EUROPEAN COMMUNITIES

MANUFACTURING SPECIFICATION FOR HIGH TEMPERATURE THERMOCOUPLES FOR USE IN NUCLEAR IRRADIATION EXPERIMENTS

by

F. MASON

1972

Joint Nuclear Research Centre
Petten Establishment - Netherlands

Irradiation Service

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ABSTRACT

This specification details the requirements for materials of construction, methods of manufacture, test procedures, certification and packing together with acceptable standards of ancillary processes which are to be employed in the production of high temperature mineral insulated thermocouple units for irradiation experiments.

KEYWORDS

THERMOCOUPLES
RHENIUM
TUNGSTEN
TECHNICAL SPECIFICATIONS
RESEARCH REACTORS
WATER COOLED REACTORS
WATER MODERATED REACTORS
IRRADIATION
IRRADIATION DEVICES
NEUTRON SPECTRA
CAPSULES

CONTENTS

DEFINITION OF TERMS

Thermocouple unit

For the purpose of this specification a thermocouple unit is defined as either:—

1. An assembly of thermocouple cable and compensating cable. The thermocouple cable has a completed hot junction, and the hot junction end of the sheath is sealed by welding, the other end of the thermo-couple cable is joined to the compensating cable by an hermetically sealed joint. The free ends of the conductors of the compensating cable terminate in and protrude from appropriate lengths of colour coded sleeving, and are sealed to the sheath to prevent the ingress of moisture.
   The assembly is illustrated diagrammatically in Fig. 1. which shows some of the other terms used in the specification.

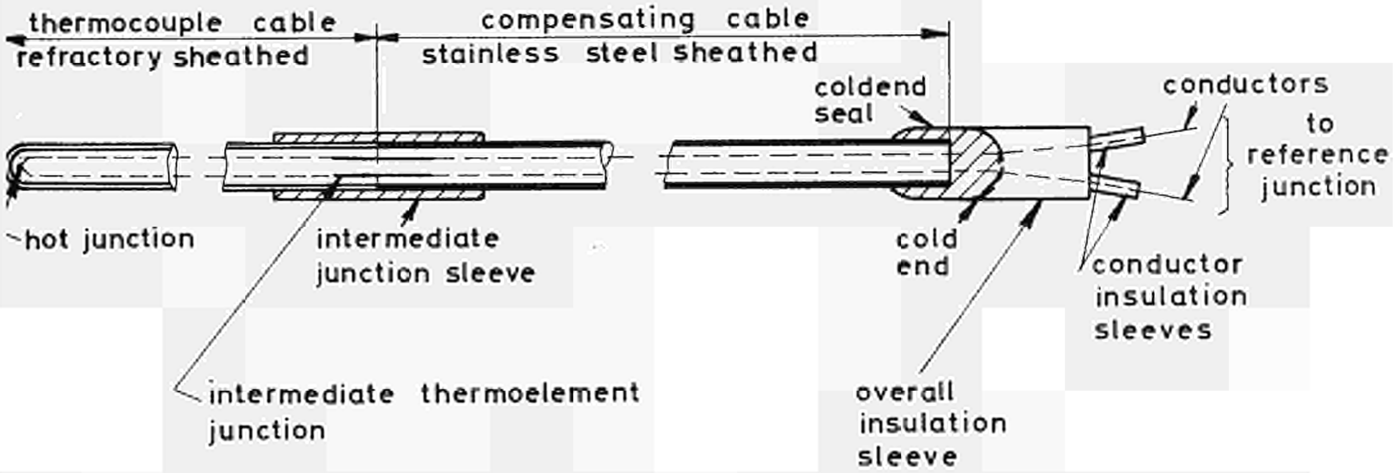


Figure 1. Thermocouple Unit. Bi-metallic, bi-insulant, bi-conductor construction

2. An assembly of continuous thermoelement wires insulated from two different enclosing sheaths by two different mineral insulants. The completed hot junction is enclosed within a refractory metal sheath, and the refractory metal sheath is joined to a stainless steel sheath by an heremetically sealed joint. The free ends of the thermoelement wires terminate in and protrude from appropriate lengths of colour coded sleeving, and are sealed to the sheath to prevent the ingress of moisture.
The assembly is illustrated diagrammatically in Fig. 2, which shows some of the other terms in the specification.

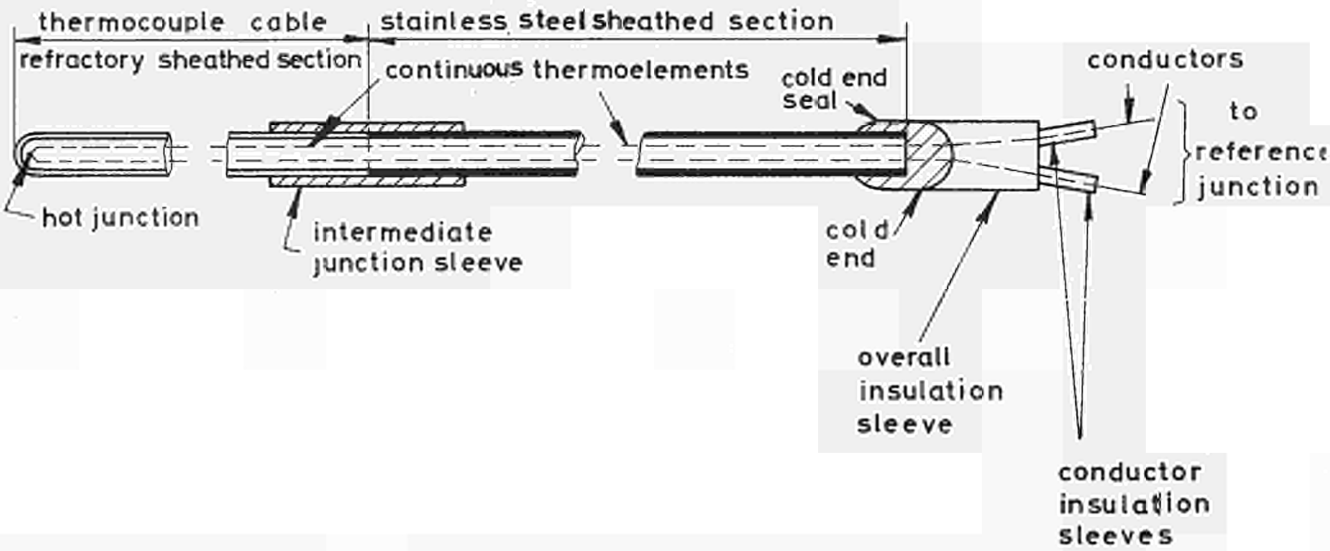


Figure 2. Thermocouple Unit. Bi-metallic, bi-insulant, continuous conductor construction

Thermocouple cable

The assembly of two dissimilar metal or alloy conductors which are insulated from each other and the enclosing sheath by a refractory mineral oxide.

Compensating cable

A flexible assembly of mineral insulated thermoelements enclosed by a metallic sheath. Over a wide range of temperature, these thermoelements have thermal-e.m.f. characteristics which are similar to the nominated thermocouple which will be used to measure temperature.

Hot junction

The junction of the two dissimilar metal conductors which respondsdirectly to temperature by generating an e.m.f. and is used to determine the temperature of a substance which is presented to it.

Reference junction

The junction of a thermocouple circuit which is maintained at a known temperature and provides the reference for determining the hot junction temperature.

Intermediate junction

The junction of the thermocouple unit where the conductors of the thermocouple cable connect to those of the compensating cable.

Cold end

That end of the thermocouple unit, most remote from the hot junction, at which the conductors of the stainless sheathed cable emerge from the protective sheath,

Customer

The customer is defined as the Technical Officer of the organisation which is producing experimental equipment, and who is responsible for ordering and specifying thermocouples for a particular application.

Contract

The purchasing order received by the manufacturer of the thermocouple units. The contract can refer to a single thermocouple unit or may be for several such units, in a variety of sizes.

Batch

Within this specification, batch defines any of the stocks of material used, which have a common certification of origin and applies equally to conductors, insulants, tubing, and extension wires.

1. SCOPE

1.1 This specification covers the requirements for materials, manufacture, testing, calibration and packing for thermocouple units which have the following characteristics:

1.1.1 The conductors of the thermocouple cable are alloys of Wolfram 5% Rhenium and Wolfram 26% Rhenium, or Wolfram 3% Rhenium and Wolfram 25% Rhenium.

1.1.2 Mineral Oxide is used as an insulant.

1.1.3 The thermoelements, insulant and hot junction are contained in a gas tight sheath.

1.1.4 The hot junction of the thermocouple is insulated from the sheath.

1.1.5 The thermocouple unit comprises a length of refractory metal sheathed thermocouple cable which is joined to a length of stainless steel sheathed cable.
The stainless steel sheathed section of unit may carry either thermoelements which are continuous from the hot junction, or lengths of the appropriate compensating wire.

1.1.6 The thermocouple unit will be installed in anirradiation experiment so that the operating temperatures of the hot and intermediate junctions will not exceed 1600°C. and 700°C. respectively, and the atmosphere during operation will be limited to inert gases. The sheath of stainless steel sheathed section of the unit must be suitable for welding or brazing into containments, and feed through seals or similar devices without sustaining damage to the sheath or conductors.

2. GENERAL CODING.

2.1. Thermocouple units produced to this specification will be given a code number in order to provide a ready reference and identification. The code number will consist of a series of digits and letters which will refer solely to the materials of the thermocouple cable.
The code employed is appended and each separate series of digits or numbers will be separated by a line, thus—

2.2 Describes the code for the refractory sheathed section of the unit.

2.2.1 The first digit in the series indicates the number of conductors in the cable.

2.2.2 The second combination will refer to the alloys which comprise the thermocouple cable conductors thus: W5Re/W26Re indicates that the conductors are Wolfram 5% Rhenium and Wolfram 26% Rhenium, and W3Re/W25Re indicates that the conductors are Wolfram 3% Rhenium and Wolfram 25% Rhenium.

2.2.3 The third combination will refer to the hot junction sheath material and the chemical symbol will be employed. For alloys this will be followed by a number and a second symbol to indicate the percentage of the secondary metal thus:-

Mo 50 Re	- denotes molybdenum 50% rhenium alloy
Mo	- denotes molybdenum
Nb	- denotes niobium
W 26 Re	- denotes Wolfram 26% Rhenium alloy
W	- denotes Wolfram.

2.2.4 The fourth combination will be a number which indicates the nominal outside diameter of the thermocouple cable in the tenths of a millimetre

2.2.5 The fifth combination will show the insulant chemical symbol, followed by a single oblique.

BeO denotes beryllium oxide insulant
MgO denotes magnesium oxide insulant.

2.2.6 The final combination will show the length of the refractory sheath from the end of the transition sleeve in cm. This combination will be followed by a double oblique to separate the unit components.

2.2.7 For illustration a complete code number for a typical thermocouple cable would be

2 — W5Re/W26Re — Mo — 13.0 — BeO / —47cm//

Two conductors of Wolfram 5% Rhenium Wolfram 26% Rhenium in a Molybdenum sheath of 1.30 mm Ø insulated by Beryllia with 47 cm of refractory sheath

2.3 Describes the code for the stainless steel sheathed section of the unit.

2.3.1 The first digit in the series indicates the number of conductors in the cable.

2.3.2 The second combination will refer to the alloys which comprise the conductors in the stainless steel sheathed section of the cable. The wire manufacturers designation will be used through-out, and will thus be one of the following:

(a) W5Re/W26Re denoting W/Re 5/26% wires
(b) W3Re/W25Re denoting W/Re 3/25% wires
(c) 405/426 denoting Hoskins compensating wires for 5/26 W/Re thermoelements.
(d) 203/225 denoting Hoskins compensating wires for 3/25 W/Re thermoelements.
(e) 300P/300N denoting Engelhard compensating wires for 3/25 W/Re thermoelements.

2.3.3 The third combination will refer to the sheath material and will normally be AC for stainless steel

2.3.4 The fourth combination will be a number which indicates the nominal outside diameter of the stainless steel section in tenths of a millimetre

2.3.5 The fifth combination will show the insulant chemical symbol and will normally be the symbol MgO for magnesia. This will be followed by a single oblique.

2.3.6 The final combination will show the length of sheathed cable from the end of the transition sleeve in cm. which completes the code number.

2.3.7 For illustration a typical thermocouple extension cable code number could be.

2 — 405/426 — Ac — 10 — MgO/ — 348 cm.

Two conductors of Hoskins 405/426 wires in a stainless steel sheath of 1.0 mm ⌀ insulated by magnesia with 348 cm. length of stainless sheath

2.4 The thermocouple unit composed of the items described in Paras 2.2 and 2.3 and joined together would be 2—W5Re/W26Re—Mo—13.0—BeO/47cm.//2—405/426—Ac—10—MgO/348cm.

3. COLOUR CODING

3.1 The manufacturer shall supply the thermocouple unit with the free ends of the compensating cable conductors terminating in a minimum length of 10 centimeters, of which at least 5 centimeters shall be suitably insulated by polyvinylchloride sleeves, the colour of these sleeves shall conform to one of the following codes:—

1	For 5/26 W-Re		
	Positive conductor	-	green
	Negative conductor	-	blue
	Overall colour	-	white
2	For 3/25 W-Re		
	Positive conductor	-	pink
	Negative conductor	-	blue
	Overall colour	-	white.

3.2 The attachment of these sleeves shall not interfere with the requirements regarding the leak tightness of the cold end seal.

4. CONDITIONS OF MANUFACTURE.

4.1 The thermocouple unit shall be produced in accordance with the sequence of operations shown in the logic diagram provided in Figure 3, and the appropriate Inspection Schedule. Pages 14 , and 39 .

4.2 No materials other than those specified in Tables 3,4,5 & 6 shall be used as any part of the thermocouple unit unless specifically requested by the customer.

4.3 Materials shall be maintained clean and free from contamination by the materials specified in paragraphs 5.1 and 5.2 at all stages of manufacture.A special clean area must be set-up within which all thermocouple manufacturing processes are carried out.

4.4 The manufacturer shall ensure that identification of materials is possible at all stages of production.

LOGIC DIAGRAM. Fig. 3

Production of Wolfram-Rhenium Alloy Thermocouples

Operation	Relevant Paragraphs
Check material specifications	4.2; 7.1; 7.2; 7.3; 8.1; 9.1; 9.2; 10.1; 13.2; 14.2; 14.3;
Check conditions of Manufacture	4.1; 4.3; 4.4; 4.5; 5.1; 5.2; 5.3; 11.1; 13.1; 14.2; 20.3;
Make dimensional tests	6.1; 6.2; 6.3; 6.4; 6.5; 15.1; 15.2;
Make sheath and conductor ductility tests	16.1; 16.2;
Check resistance of conductors	18.1;
Make hot junction and sheath closure	11.1; 12.1; 12.2; 12.3; 12.4; 13.1; 13.2; 13.3; 17.1; 17.2; 17.3;
Radiograph hot junction	19.1;
Make intermediate junction and temporary cold seal	14.1; 14.2; 14.3; 14.4; 14.5; 17.1; 17.2; 17.3;
Radiograph intermediate junction	19.2;
Test intermediate junction	20.5; 20.6; 16.2.2; 16.2.3; 20.3; 20.4.
Calibrate thermocouple unit	20.1; 20.2; 20.3; 20.4; 20.5; 20.6;
Make hot insulation test	17.1; 17.2; 17.3;
Final cold seal and sheath integrity tests	10.1; 3.1; 3.2; 21.1; 21.2; 22.1; 22.2; 22.3; 22.4;
Final cleaning and packing	23.1; 24.1; 24.2; 24.3; 24.4; 25.1; 25.2; 25.3; 26.1; 2.1; 2.2; 2.3; 2.4; 27

4.5 The manufacturer shall ensure that adequate records of all tests are made at the time of the test, and that these records are available for inspection by the customer for a minimum period of two years after delivery of the units.

5. PRECAUTIONS AGAINST CONTAMINATION.

5.1 Finished units shall be free from surface contamination (in the form of dust, adherent film, or embedded particles) by any of the materials listed below.

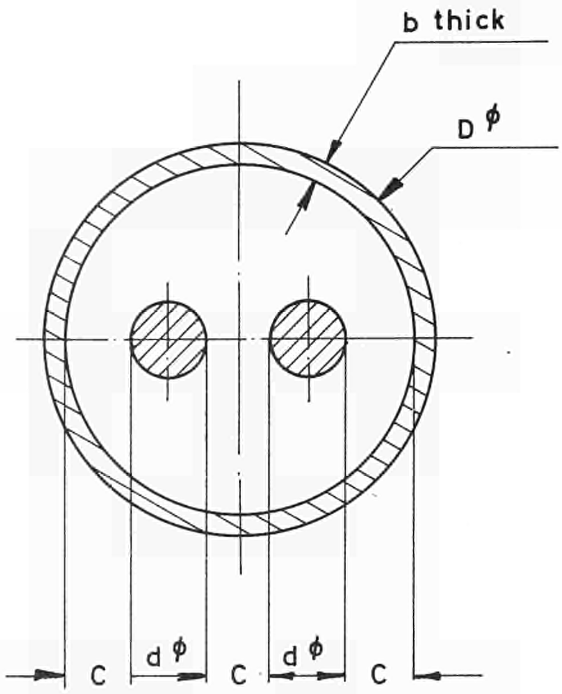
Ag	Fe	Fissile or Fertile materials
Al	Hg	
As	I	Chlorides, and rare earths.
B	Mg	
Ba	K	
Bi	Pb	
C	S	
Ca	Si	
Cd	Sb	
Cl	Sn	
Cu	Zn	
F		

5.2 During any operation which involves heat treatment, the manufacturer shall ensure that adequate precautions are taken to exclude any environment which may cause embrittlement to the sheath or conductors.

5.3 It is of particular importance that the materials are not exposed to environments of high moisture content, during storage and/or processing.

6. DIMENSIONS.

6.1 The thermocouple cable shall be manufactured to the dimensions detailed in Figure 4, and Table 1, Page 16.

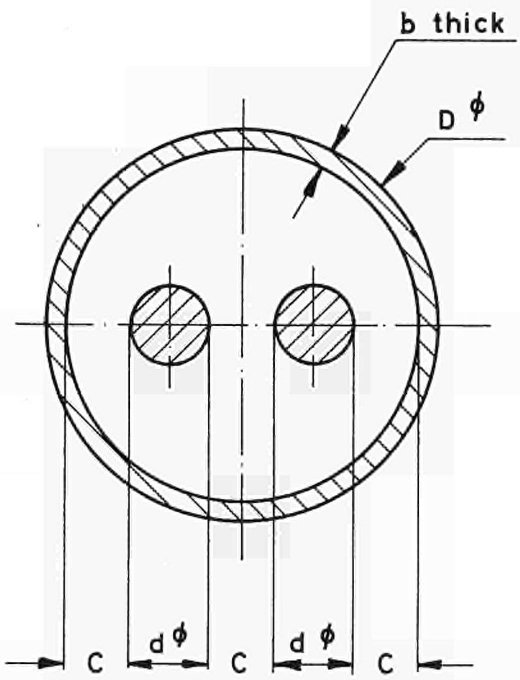


Cross-section of thermocouple cable

Figure 4

TABLE 1 THERMOCOUPLE CABLE DIMENSIONS. in mm.

Outside Diameter D		Sheath Thickness b		d Wire Diameter		C Insulant Thickness
min	max	min	max	min	max	min
1.25	1.35	0.16	0.25	0.16	0.25	0.09



Cross-section of compensating cable

Figure 5

Table 2. COMPENSATING CABLE DIMENSIONS in mm.

Outside Ø D		Sheath Thickness b min.	Insulation Thickness c min.	Conductor Ø d	
min	max			min	max
0.98	1.02	0.110	0.100	0.130	0.210
1.28	1.32	0.150	0.120	0.230	0.310
1.58	1.62	0.190	0.140	0.290	0.380

6.3 The cross section of the conductors shall be substantially circular; both maximum and minimum must lie within the limits of Tables 1 and 2.

6.4 The completed hot junction must conform to Para 12.1 and Fig. 7, Pages 23 and 25.

6.5 The intermediate junction must conform to Para 14.1 and Fig. 8, Page 25.

7. CONDUCTORS.

7.1 The conductors of the thermocouple cable shall be free from laminations and shall be manufactured from material having nominal compositions as detailed in Table 3, Page 18.

Table 3. THERMO-ELEMENT CONDUCTORS.

Material	Composition Nominal	Resistance $\mu\Omega$-cm at 20°C	Associated Specification
Tungsten 5% Rhenium	Tungsten 95% Rhenium 5%	18.0	N.B.S. mono-graph 40 table 95
Tungsten 26% Rhenium	Tungsten 74% Rhenium 26%	31.0	N.B.S.mono-graph 40 table 95
Tungsten 3% Rhenium	Tungsten 97% Rhenium 3%	9.7	N.B.S. mono-graph 40 table 95
Tungsten 25% Rhenium	Tungsten 75% Rhenium 25%	27.9	N.B.S. mono-graph table 95

7.2 The wires in the stainless steel sheathed section of the thermocouple unit may be one of the three following alternatives.

7.2.1 Compensating types of similar thermoelectric properties to the thermoelement wires up to 700°C.

7.2.2 W-Re wires of the same composition as the wires which are connected at the hot junction; and which are joined to the refractory sheathed section wires at the intermediate junction.

7.2.3 W-Re wires which are continuous from the hot junction to the cold end of the thermocouple unit.

7.3 If compensating cable is used the thermocouple manufacturer must supply certified temperature to e.m.f. details over the range 0 to 700°C for the particular compensating wires used, and precise measured deviation from the supplied curve at 400°C for each thermocouple.

See also Para. 20.3

8. INSULANT.

8.1 The insulants used shall conform to those specified in Table 4, and will be BeO in the refractory sheathed section and MgO in the stainless steel sheathed section.

Table 4. INSULATING MATERIALS.

Material	Composition	
Beryllia (BeO)	Beryllium Oxide	99.9% (min)
	Boron	0.001% (max)
	Cadmium	0.001% (max)
	Aluminium	0.01% (max)
	Calcium	0.05% (max)
	Carbon	0.001% (max)
	Ferric Oxide	0.001% (max)
	Magnesium	0.05% (max)
	Potassium	0.002% (max)
	Sodium	0.001% (max)
	Silicon Dioxide	0.002% (max)
Magnesia (MgO) (fused and crushed)	Magnesium Oxide	99.4% (min)
	Aluminium Oxide	0.60% (max)
	Silicon Dioxide	0.20% (max)
	Calcium Oxide	0.05% (max)
	Iron Oxides	0.1% (max)
	Chlorides	0.005% (max)
	Vanadium Pentoxide	0.003% (max)
	Sulphur	0.001% (max)
	Carbon	0.001% (max)
	Boron	0.001% (max)
	Cadmium	0.001% (max)

9. SHEATH.

9.1 Sheath materials shall be in the form of continuous seamless tubes, smooth and free from porosity, cracks and inclusions. The total reduction of wall thickness arising from any such defect and including surface embrittlement and oxidation shall not exceed 20% of the nominal wall thickness.

9.2 The material of the sheath shall conform to the compositions specified in Table 5, niobium is the preferred material for the refractory sheathed section of the unit.

Table 5 SHEATH MATERIALS.

Niobium	Carbon Oxygen Nitrogen Hydrogen	$<$100 p.p.m. $<$100 p.p.m. $<$100 p.p.m. $<$10 p.p.m.
	Molybdenum Titanium Vanadium Wolfram Zirconium	total $<$3500 p.p.m. No single element in excess of 1000 p.p.m.
	Tantalum	$<$1500 p.p.m.
	Aluminium Chromium Copper Iron Manganese Nickel Silicon	total $<$500 p.p.m. No single element in excess of 150 p.p.m.
	Cadmium Magnesium Lead Tin Zinc	total $<$150 p.p.m.
	Boron Hafnium Niobium	10 p.p.m. 300 p.p.m. Balance

Table 5 SHEATH MATERIALS.(CONTD.)

50% Molybdenum 50% Rhenium	Carbon	<5 p.p.m.
	Oxygen	<5 p.p.m.
	Aluminium	<10 p.p.m.
	Calcium	<5 p.p.m.
	Chromium	<10 p.p.m.
	Copper	<5 p.p.m.
	Iron	<50 p.p.m.
	Magnesium	<5 p.p.m.
	Manganese	<5 p.p.m.
	Sodium	<5 p.p.m.
	Nickel	<10 p.p.m.
	Silicon	<10 p.p.m.
	Tin	<5 p.p.m.
	Molybdenum	45-55%
	Rhenium	Balance
Tungsten 74% Rhenium 26%	Minimum of 99.9% purity	
Molybdenum	Carbon	<0.005%
	Oxygen	<0.005%
	Hydrogen	<0.001%
	Nitrogen	<0.001%
	Silicon	<0.010%
	Nickel	<0.003%
	Cobalt	<0.001%
	Iron	<0.010%
	Aluminium	<0.004%
	Boron	<0.001%
	Halogens	<0.001%
Tungsten	Carbon	<0.005%
	Oxygen	<0.005%
	Nickel	<0.003%
	Iron	<0.010%
	Aluminium	<0.004%
	Boron	<0.001%
	Halogens	<0.001%

Table 5 SHEATH MATERIALS (CONTD.)

18/8/1 Nb. Stainless Steel	Chromium 17-19% Nickel 9-12% Niobium ≧10 x C Carbon 0.8% max Manganese 2.0% max Silicon 1.0% max Phosphorous 0.04% max Sulphur 0.03% max Iron Balance	Types: A.I.S.I.347 B.S.En 58F D.I.N. Werk-stoff No. 1-4541 A.F.N.O.R. Z6 CN Nb17-11
18/8/1 Ti. Stainless Steel	Chromium 16-18% Nickel 8-11% Titanium ≧5xC Manganese 2% max Silicon 1% max Phosphorous 0.04% max Sulphur 0.03% max Carbon 0.8% max	AISI 321
18/8 Low Carbon Stainless Steel	Chromium 16-18% Nickel 10-14% Molybdenum 2-3% Manganese 2% max Silicon 1% max Phosphorous 0.45% max Sulphur 0.03% max Carbon 0.03% max	A.I.S.I. 316L

10. SEALING COMPOUNDS.

10.1 The materials used for sealing the cold end of the cable shall conform to those specified in Table 6 overleaf.

Table 6 SEALING COMPOUNDS.

Material	Temperature limitation in use
*Araldite AY 105 with HY 972	80^oC
*Araldite AY 111 with HY 111	80^oC
**Thermofit Sleeving (Dualwall irradiated plastic)	According to type used
†Swifts K 9903 hot melt adhesive	80^oC
††Rilsan (To be used only when prior agreement is obtained from the customer)	100^oC

*Trade names of C.I.B.A. (A.R.L.) Ltd. Duxford, Cambridge, England.

**Trade name of Raychem U.K.Ltd. Manningtree, Essex,England.

†Trade name of Swift & Co., Spelthorne Lane, Ashford, Middlesex,England Available from Swift et Comp. Rue d'l' Vallee aux Boeufs, 41 Blois, France

††Trade name of Aquitaine Organico, 16 Rue d'Artois, Paris.

11. PRECAUTIONS DURING HEAT TREATMENT.

11.1 Any operations which involve heating the thermocouple unit to a temperature above 300^oC shall be conducted in either a vacuum, or a helium or an argon atmosphere of such a quality that sheath or conductor oxidation or embrittlement is avoided.

12. HOT JUNCTION MANUFACTURE. (Figs 6&7).

12.1 The thermoelement wires must be arranged as shown in Fig. 6 before completing the hot junction, and conform with Fig. 7 after completion, Page 25.

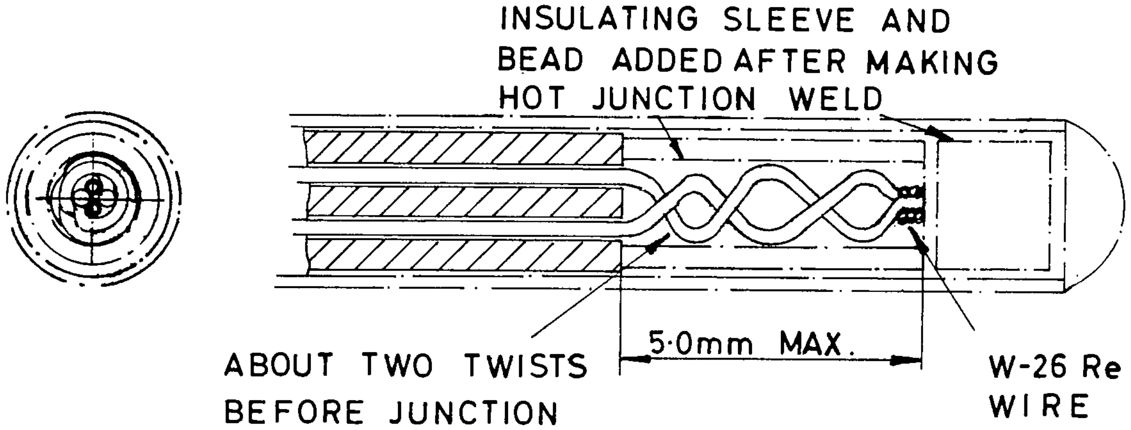


FIGURE 6 ARRANGEMENT OF HOT JUNCTION.

12.2 It will be preferred for the two thermoelements to be twisted together, bound with a single thin strand of W 26% Re wire, and the four ends fused together by an inert gas arc weld. It is essential that the area within the sheath, and adjacent to the hot junction is scrupulously cleaned, prior to the introduction of the hot junction insulation, and completion of the sheath closure weld.

12.3 Niobium may be used as a braze metal to join the conductors at a temperature below their fusion point. The atmosphere during this operation shall be either vacuum, or helium or argon as specified in Paragraph 11.1.

12.4 Where the hot junction consists of jointed conductors which protrude from preformed beads, the manufacturer shall take all precautions to ensure that the junction cannot short circuit to the sheath. A mineral oxide sleeve must be used as an insulator. The material of the insulator sleeve shall be to the same specifications as the preformed insulating beads.

13. <u>SHEATH CLOSURE</u>.

13.1 The sheath closure shall be made by fusion of the metals, the process and atmosphere being such as to avoid oxidation or embrittlement. It is advantageous to back fill to about 1 atmosphere with He during the final sheath closure operation. The He must have a combined H_2O plus O_2 concentration of < 30 p.p.m.

13.2 When a separate end plug is used for sheath closure the plug shall be of the same composition and to the same specification as that of the sheath material.

13.3 The closure shall be inspected and shall have the same freedom from defects as specified in Paragraph 9.1., and must be to the dimensions of Fig. 7, Page 25.

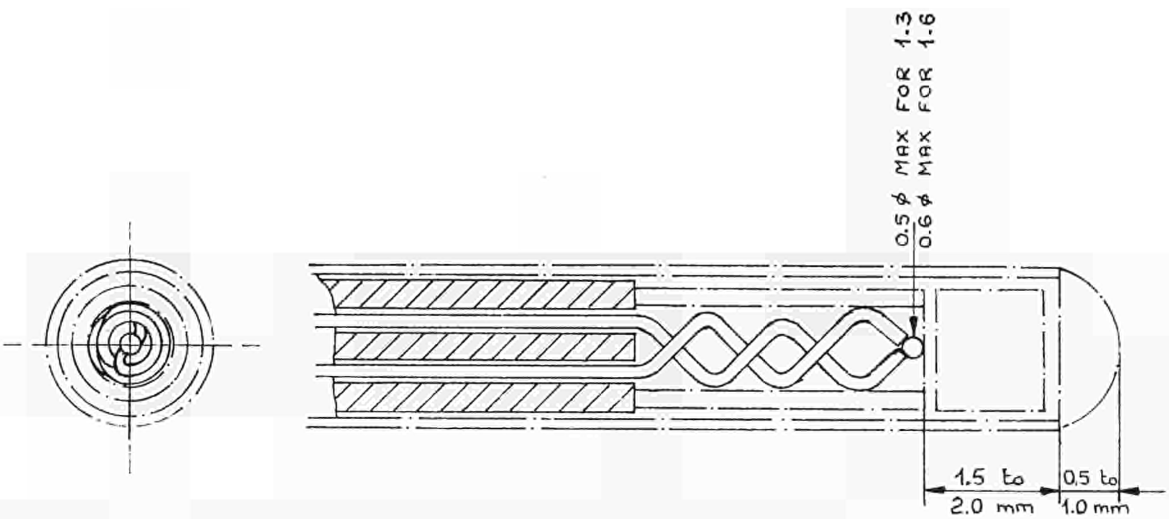


FIGURE 7 ARRANGEMENT OF COMPLETED HOT JUNCTION.

14. INTERMEDIATE JUNCTION MANUFACTURE.

14.1 The intermediate junction must conform to the dimensions of Fig. 8, below for Para 7.2.1 and Para 7.2.2 construction. With continuous wires as Para 7.2.3., reference to wire jointing is ignored, but information on the transition sleeve still applies.

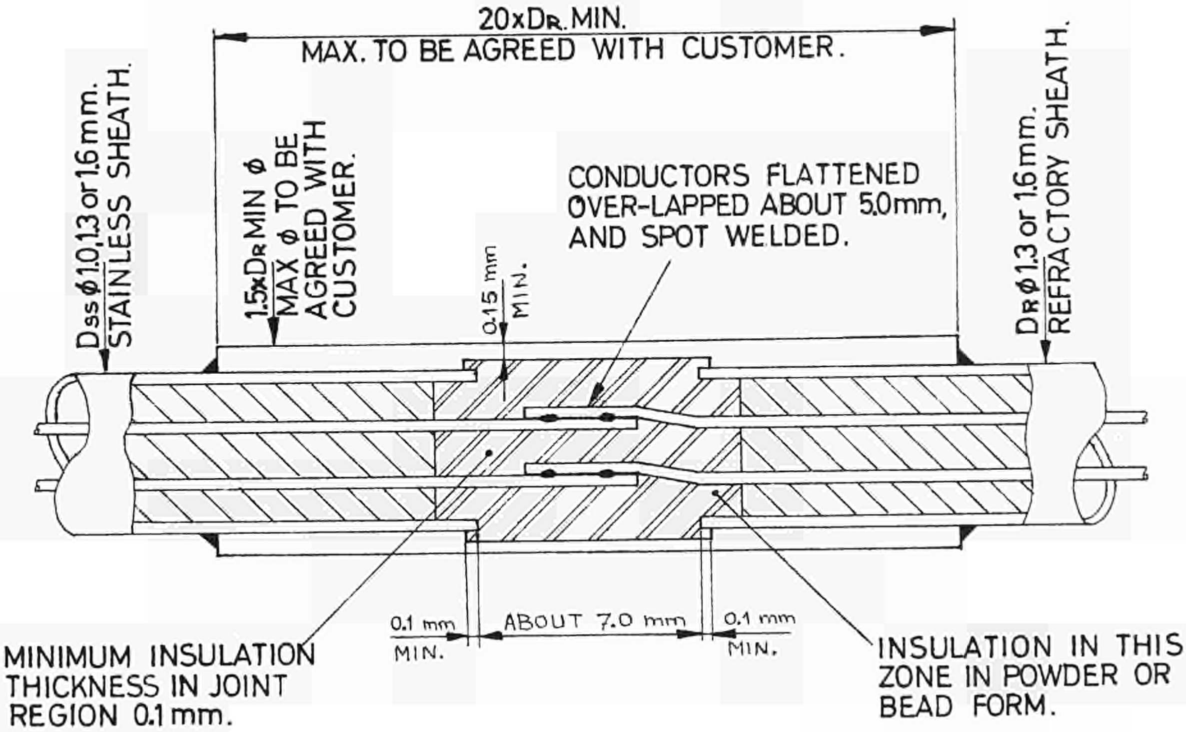


FIGURE 8 ARRANGEMENT OF INTERMEDIATE JUNCTION.

14.2 The thermocouple conductors and the compensating cable conductors shall be joined by spot welding and shall be insulated as necessary by a mineral oxide insulator having the same material specification as the insulant of the compensating cable. It is advantageous to back fill to about 1 atmosphere with He during the final sheath closure operation. The He must have a combined O_2 plus H_2O concentration of 30 p.p.m.

14.3 The sleeve used to join the sheath of the thermocouple cable to the sheath of the compensating cable shall be of the same material and specification as the sheath of the compensating cable.

14.4 The joint between the sleeve and the refractory metal sheath of the thermocouple unit shall be inert gas arc welded.
The joint betweenthe compensating cable sheath and the sleeve shall be inert gas are welded.

14.5 The sleeve and its associated joints shall be inspected and shall have the same freedom from defects as specified in Paragraph 9.1, and must be suitable for operation at 700°C.

15. DIMENSIONAL TESTS.

15.1 A sample cut from each length of compensating cable and thermocouple cable shall be examined to ensure that sheath, conductor and insulant dimensions conform to the dimensions specified in Paragraphs 6.1, 6.2 and 6.3.
The prefered diameter is 1.6 mm.

15.2 Where the thermocouple cable consists of an unworked refractory sheath containing preformed insulating beads the dimensions of sheath, hot junction and conductors shall conform to Fig. 8 and the following data shall be recorded:

Measured inside and outside dimensions of refractory sheath and material.

Measured maximum and minimum diameter of each conductor.

Measured maximum and minimum diameter of the preformed insulating beads.

It is essential with this type of construction that a BeO disc is placed between the hot junction and the sheath closing weld to preclude any possibility of the hot junction contacting the sheath during operation.

16. DUCTILITY TESTS

16.1 Conductors

A sample 5 cms. long shall be taken from each batch of all conductor wires which will be used for the contract. The sample shall be bent through a right arc and back five times, the radius of bend being four times the conductor diameter. All wire from that batch shall be rejected if the sample breaks.

16.2 Sheaths

16.2.1 Compensating cable

A sample 25 cms. long shall be taken from each basic reel of compensating cable that is required for the contract, and sealed at both ends. The sample shall be coiled six times round a mandrel, which has a diameter of four times the nominal diameter of the compensating cable. The sample shall be tested by subjecting it to a sheath integrity test as described in paragraph 22.2. The cable reel shall be rejected if the sample fails this test.

16.2.2. <u>Thermocouple cable</u>.

Niobium sheathed cable is subject to the following test.

One thermocouple unit of each order, or production group of thermocouple units manufactured from the same batch materials, shall have the niobium sheath bent 90° round a mandrel, which has a diameter of sixteen times the nominal sheath diameter and straightened. This test is to be preceded and followed by a helium leak test to paragraph 22.2, a loop resistance test to paragraph 18, and an insulation resistance test to paragraph 17, at room temperature. All results are to be recorded. The test detailed in paragraph 16.2.3, could, with advantage, be carried out on the same thermocouple.

16.2.3. <u>Intermediate junction</u>.

The stainless steel sheathed cable is subject to the following test.

One thermocouple unit of each order or production group of thermocouple units manufactured from the same batch materials, shall be subjected to a bending moment applied to the stainless steel sheath, at a point 20 mm. distant from the end of the intermediate junction sleeve. The applied force must be sufficient to move the cable through an angle of 30° in two directions in the same plane. This test is to be preceded and followed by a helium leak test to paragraph 22.2, a loop resistance test to paragraph 18, and an insulation resistance test to paragraph 17, at room temperature. All results are to be recorded the test detailed in paragraph 16.2.2. could, with advantage be carried out on the same thermocouple unit if it is required.

17. INSULATION RESISTANCE TESTS.

17.1 The completed thermocouple unit shall have an insulation resistance in accordance with the following table.

Table 7 Insulation Resistance

Measurement between	Temperature of test (^{o}C)	Test Voltage	Minimum acceptable insulation resistance (Ohms)
Conductors	20 ± 5	100 ± 20	5×10^{10}
to sheath;	700 ± 20	9 ± 1	5×10^{6}

17.2 When testing the thermocouple unit at 700°C at least 75% of the refractory sheathed section of the unit shall be subjected to the specified temperature, the intermediate junction shall not be subjected to a temperature exceeding 500°C during this test. The precautions of paragraph 11 apply during this test.

17.3 The complete thermocouple unit excluding the cold seal shall be heated to 400°C and the insulation resistance measured between conductors and sheath at a test voltage of 9V $\pm$ 1 V.
The product of insulation and length shall be not less than 10^7 Ohm metres i.e.
Thermocouple length (metres) x insulation resistance (Ω)
1×10^7 Ohm metres.

18. LOOP RESISTANCE TEST.

18.1 The loop resistance (R_L) of the completed thermocouple unit shall be measured at an ambient temperature of approximately 20°C, the resistance value obtained shall not exceed the sum of the component conductor resistance by more than 1.0% i.e.

$$R_L = 1.010 \left[R_1 + R_2 + R_3 + R_4 \right]$$

where R_1 = resistance of the positive conductor of the thermocouple cable

R_2 = resistance of the negative conductor of the thermocouple cable

R_3 = resistance of the positive conductor of the compensating cable

R_4 = resistance of the negative conductor of the compensating cable

The resistance of the individual wires must lie within the values given in table 8.

Table 8 CONDUCTOR RESISTANCE.

NOMINAL CABLE Ø mm	CONDUCTOR TYPE	CONDUCTOR DIAMETER		RESISTANCE Ω/m at20°C	
		min	max	max	min
1.3	W5Re	0.16	0.25	8.92	3.66
	W26Re	0.16	0.25	15.38	6.31
1.6	W5Re	0.20	0.30	5.73	2.56
	W26Re	0.20	0.30	9.89	4.40
1.0	405	0.13	0.21	22.40	8.58
	426	0.13	0.21	24.7	9.45
1.3	405	0.23	0.31	7.19	3.97
	426	0.23	0.31	7.90	4.36
1.6	405	0.29	0.38	4.50	2.62
	426	0.29	0.38	4.95	2.88
1.3	W3Re	0.16	0.25	4.80	1.97
	W25Re	0.16	0.25	13.82	5.66
1.6	W3Re	0.20	0.30	3.09	1.38
	W25Re	0.20	0.30	8.89	3.96
1.0	300P	0.13	0.21	32.50	12.40
	300N	0.13	0.21	11.61	4.44
1.3	300P	0.23	0.31	10.40	5.76
	300N	0.23	0.31	3.71	2.05
1.6	300P	0.29	0.38	6.52	3.80
	300N	0.29	0.38	2.22	1.35

Table 8. CONDUCTOR RESISTANCE (CONTD.)

NOMINAL CABLE Ø	CONDUCTOR TYPE	CONDUCTOR DIAMETER		RESISTANCE Ω/m at 20°C	
		min	max	max	min
1.0	203	0.13	0.21	59.0	22.55
	225	0.13	0.21	22.4	8.59
1.3	203	0.23	0.31	18.9	10.4
	225	0.23	0.31	7.18	3.96
1.6	203	0.29	0.38	11.8	6.88
	225	0.29	0.38	4.51	2.62

19. RADIOGRAPHY.

19.1 The end closure, hot junction and adjacent 10 cms of the thermocouple shall be radiographed in two planes mutually at right angles and perpendicular to the axis of the conductors. The quality of the radiograph shall be adequate to enable an assessment of the following (i) compliance with the dimensional requirements of paragraphs 6.1, 6.4 and 12.1 (ii) cracks, notches, crevices, voids or inclusions in the conductors, junction, sheath and end closure.

Failure to meet the requirements of paragraphs 6.1, 6.4 and 12.1 or the existence of any defect as stated in (ii) above, shall cause rejection of the thermocouple unit.

19.2 Any intermediate junction shall be radiographed in a similar manner to that specified in paragraph 19.1. The field of the radiograph shall include a minimum length of 5 cms on either side of the joint. The quality of the radiograph shall be adequate to indicate defects as listed in paragraph 19.1 and in addition, any lack of fusion at the sleeve enclosing the sheaths. Any such defect shall cause rejection of the thermocouple unit.

20. CALIBRATION.

Requirement and Method.

20.1 One thermocouple unit of each order or production group of thermocouple units, manufactured from the same batch materials must be calibrated, at three points specified by the customer from the following list of temperature ranges by comparison with a secondary standard thermocouple which has been independently calibrated at the six mentioned fixed points in the I.P.T.S. 68.

$95^{o}C$ to $105^{o}C$ (B.P. of H_2O $100^{o}C$)
$412^{o}C$ to $428^{o}C$ (F.P. of Zn $419.58^{o}C$)
$620^{o}C$ to $640^{o}C$ (F,P. of Sb $630.74^{o}C$)
$950^{o}C$ to $970^{o}C$ (F.P. of Ag $961.93^{o}C$)
$1050^{o}C$ to $1080^{o}C$ (F.P. of Au $1064.43^{o}C$)
$1535^{o}C$ to $1575^{o}C$ (F.P. of Pd $1554^{o}C$)

The secondary standard thermocouple, together with the thermocouple unit to be tested, are to be inserted into an iso-thermal region of the calibration furnace. The minimum length of thermocouple unit which is to be immersedin the iso-thermal region is 50 mm. or 25% of the refractory sheathed section, whichever is the greater. The output of the secondary standard thermocouple shall be taken as indicating true temperature of the iso-thermal region when referred to the secondary standard calibration curve.

20.2 Calibration Accuracy.

The manufacturer of thermocouple units shall obtain a certificate of wire calibration from the producer of all the thermocouple cable conductors.
This certificate will show the identities of the wire pairs and the deviation of the particular batch from Hoskins November 1962 Table for W-Re 5/26 wire and 405/426 compensating wires; and Engelhards Technical Bulletin, Vol. XI No. 1 June 1970, for W-Re 3/25 wires and 300P/300N, or 203/225 compensating wires.
These tables are on pages 33 and 34.

"The thermocouple unit when calibrated as specified in paragraph 20.1, shall agree with the appropriate table within 1.5%, in the temperature ranges, $300^{\circ}C$ to $1550^{\circ}C$, with a maximum acceptable measuring inaccuracy of 0.5%."

Table 9

Note: CARE SHOULD BE TAKEN IN THE USE OF THESE TABLES, AS THEY MAY BE SUBJECT TO REVISION.

Reference Tables* for Tungsten 5% Rhenium vs Tungsten 26% Rhenium Thermocouples

Reprinted by permission of Hoskins Manufacturing Co., DETROIT, Michegan, U.S.A., from whom certificated matched pairs of thermo-element and compensating wires may be obtained. Electromotive force in millivolts. Temperatures in $^{\circ}C$. Reference junction at 0 $^{\circ}C$.

°C	0	10	20	30	40	50	60	70	80	90	100
0	0.000	0.103	0.234	0.369	0.506	0.645	0.788	0.933	1.080	1.230	1.382
100	1.382	1.535	1.689	1.846	2.003	2.163	2.324	2.487	2.652	2.820	2.988
200	2.988	3.158	3.330	3.504	3.679	3.857	4.036	4.217	4.400	4.583	4.768
300	4.768	4.954	5.142	5.330	5.518	5.707	5.896	6.085	6.275	6.465	6.655
400	6.655	6.845	7.036	7.227	7.418	7.610	7.802	7.995	8.187	8.380	8.573
500	8.573	8.767	8.960	9.154	9.347	9.541	9.734	9.928	10.121	10.315	10.508
600	10.508	10.702	10.896	11.091	11.285	11.480	11.674	11.868	12.063	12.257	12.450
700	12.450	12.644	12.837	13.030	13.223	13.415	13.608	13.800	13.991	14.183	14.374
800	14.374	14.565	14.755	14.945	15.135	15.324	15.513	15.702	15.890	16.078	16.266
900	16.266	16.453	16.640	16.826	17.012	17.198	17.383	17.568	17.753	17.937	18.120
1000	18.120	18.304	18.488	18.671	18.854	19.036	19.218	19.400	19.582	19.763	19.944
1100	19.944	20.124	20.304	20.484	20.663	20.842	21.020	21.197	21.374	21.559	21.724
1200	21.724	21.899	22.072	22.244	22.416	22.587	22.757	22.925	23.093	23.260	23.424
1300	23.424	23.588	23.751	23.914	24.076	24.237	24.397	24.557	24.716	24.874	25.033
1400	25.033	25.191	25.348	25.505	25.661	25.817	25.972	26.126	26.279	26.431	26.583
1500	26.583	26.734	26.885	27.035	27.185	27.335	27.484	27.633	27.782	27.930	28.078
1600	28.078	28.226	28.373	28.520	28.666	28.812	28.957	29.101	29.244	29.387	29.528
1700	29.528	29.669	29.809	29.949	30.089	30.229	30.368	30.507	30.645	30.784	30.922
1800	30.922	31.061	31.199	31.337	31.474	31.612	31.750	31.887	32.024	32.161	32.298
1900	32.298	32.435	32.571	32.706	38.841	32.975	33.108	33.240	33.372	33.502	33.632
2000	33.632	33.762	33.891	34.020	34.149	34.277	34.405	34.533	34.661	34.788	34.915
2100	34.915	35.041	35.167	35.291	35.414	35.535	35.654	35.769	35.881	35.987	36.089
2200	36.089	36.191	36.278	36.368	36.455	36.538	36.619	36.698	36.776	36.853	36.929
2300	36.929	37.004	37.078								

*Adopted 23rd. November 1962, by Hoskins Manufacturing Co.

Table 10

Note: CARE SHOULD BE TAKEN IN THE USE OF THESE TABLES, AS THEY MAY BE SUBJECT TO REVISION.

Reference Tables* for Tungsten 3% Rhenium vs Tungsten 25% Rhenium Thermocouples

Reprinted by permission of:- Engelhard Industries Inc. Research Division, NEWARK, New Jersey, U.S.A., from whom certificated matched pairs of thermo-element and compensating wires may be obtained.
Electromotive force in millivolts. Temperature in °C. Reference junction at 0 °C.

°C	0	10	20	30	40	50	60	70	80	90
0	0.000	0.978	0.200	0.305	0.415	0.528	0.644	0.765	0.888	1.015
100	1.145	1.278	1.415	1.554	1.696	1.840	1.988	2.138	2.290	2.445
200	2.602	2.761	2.923	3.087	3.253	3.420	3.590	3.762	3.935	4.110
300	4.286	4.465	4.644	4.825	5.008	5.192	5.377	5.563	5.751	5.940
400	6.129	6.320	6.512	6.705	6.899	7.093	7.288	7.484	7.681	7.879
500	8.077	8.275	8.475	8.675	8.875	9.076	9.277	9.479	9.681	9.883
600	10.086	10.289	10.492	10.695	10.899	11.103	11.307	11.511	11.716	11.920
700	12.124	12.330	12.534	12.738	12.943	13.147	13.352	13.557	13.761	13.966
800	14.170	14.374	14.578	14.782	14.986	15.190	15.393	15.597	15.800	16.003
900	16.211	16.414	16.617	16.819	17.021	17.223	17.424	17.625	17.826	18.026
1000	18.226	18.426	18.625	18.824	19.023	19.221	19.419	19.616	19.813	20.009
1100	20.206	20.401	20.597	20.791	20.986	21.180	21.373	21.566	21.759	21.951
1200	22.143	22.334	22.525	22.715	22.905	23.094	23.283	23.471	23.659	23.847
1300	24.033	24.220	24.406	24.591	24.776	24.961	25.145	25.328	25.511	25.693
1400	25.875	26.057	26.238	26.418	26.598	26,777	26.956	27.134	27.312	27.490
1500	27.666	27.842	28.018	28.193	28.368	28.542	28.715	28.888	29.061	29.233
1600	29.404	29.575	29.745	29.914	30.083	30.252	30.419	30.587	30.753	30.919
1700	31.085	31.249	31.413	31.577	31.740	31.902	32.063	32.224	32.384	32.543
1800	32.702	32.860	33.017	33.174	33.330	33.485	33.639	33.792	33.945	34.097
1900	34.248	34.398	34.547	34.695	34.843	34.989	35.135	35.279	35.423	35.566
2000	35.707	35.848	35.987	36.126	36.263	36.400	36.535	36.669	36.801	36.933
2100	37.063	37.192	37.320	37.446	37.571	37.694	37.816	37.937	38.056	38.174
2200	38.290	38.404	38.517	38.628	38.738	38.846	38.951	39.056	39.158	39.258
2300	39.357	39,453	39.547	39.640	39.730	39.818	39.903	39.987	40.068	40.147
2400	40.223									

* Adopted June 1970, by Engelhard Industries Inc.

20.3 Intermediate junction e.m.f. (comp.wires)

Where the conductors of the compensating cable are not identical to the thermocouple conductors, the following test shall be made. The hot and cold junctions shall be maintained at $0^{o}C$ whilst the intermediate junction is heated to $395^{o}C$ to 405 ^{o}C. The emf. measured at the cold junction shall not exceed 0.15 mv. at this temperature, and must be noted on the inspection certificate as a deviation from the supplied temperature/ e.m.f. Table.

20.4 Intermediate junction e.m.f.(continuous wires).

Where the wires in the refractory and stainless steel sheaths are identical in composition, the intermediate junction shall be heated to $390^{o}C$ to $410^{o}C$ whilst the hot and cold junctions are maintained at $0^{o}C$. The emf. measured at the cold junction shall not exceed 0.05 mv, at this temperature, and must be noted on the inspection certificate.

20.5 Alternatives.

The customer may accept the calibration data supplied by the manufacturer of the conductor wire as an alternative to the requirements of paragraph 20.1 and 20.2 for units with unworked refractory sheaths. The manufacturer must apply to the customer if this concession is required.

20.6 Damage.

Damage to the sheath during the calibration procedure shall be limited to that specified in paragraph 9.1.

21. SHEATH INTEGRITY TESTING (Water immersion).

21.1 Unless otherwise specified by the customer the complete thermocouple unit, but excluding the cold end seal, shall be immersed for four hours in water to which has been added a surface tension reducing agent (see Appendix 1) so that the surface tension does not exceed 35 dynes/cm. The insulation resistance of the unit shall be not less than 5×10^{10} ohms at 100 $\pm$ 20 V test voltage when measured within one hour of removal from the water. During this test, heating of the thermocouple unit is not permitted.

21.2 The customer may specify at the time of ordering of the thermocouple that either of the tests detailed in paragraphs 22.2 and 22.3 shall be applied as an alternative to paragraph 21.1. In which case the complete thermocouple but excluding the cold end seal shall be pressurized.

22. SHEATH INTEGRITY TESTING (Helium leak).

22.1 If either of the two following tests are necessary the customer will specify which test is required when the thermocouple unit is ordered.

22.2 The hot junction and the adjacent 10 cms of thermocouple shall be subjected to an external helium pressure of 100 kg cm^{-2} $\pm$ 10 Kg cm^{-2} for a period of 15 minutes. The pressurized length shall then be wiped with a cloth soaked in acetone, and immediately inserted into an evacuation chamber which is connected to a helium mass spectrometer. The chamber shall be evacuated and tested for helium. An indicated source of helium greater than 6×10^{-9} atmos. mL sec^{-1} shall be cause to reject the thermocouple. The sensitivity of the mass spectrometer and evacuation chamber assembly shall be confirmed before and after this test and shall be not less than 3×10^{-9} atmos. mL. $sec.^{-1}$.

22.3 The hot junction and the adjacent 10 cms of thermocouple shall be subjected to an external helium pressure of 100 Kg cm^{-2} $\pm$ 10 Kg cm^{-2} for a period of 15 minutes. The thermocouple shall be wiped with an acetone soaked cloth and immediately plunged into alcohol. There shall be no bubbles evolved during the succeeding two minutes.

22.4 The customer may request that the refractory sheathed section of the thermocouple unit, and the intermediate junction are subjected to a dye penetrant test.

23. FINAL CLEANING.

23.1 When manufacturing and testing is completed the thermocouple units shall be cleaned by total immersion for 2 hours in DECON 90* or other cleansing agent agreed with the customer. The unit shall be washed in fresh demineralised water to remove the cleansing agent. Thereafter it shall be handled with clean cotton gloves. When the unit has been dried it shall be packaged as subsequently detailed.

24. PACKING.

24.1 The unit shall be contained in a sealed polythene bag. The bag shall contain radiographs, the calibration and test data, the inspection certificate, and an identification label.

24.2 The refractory sheathed section of the unit must remain straight.

24.3 The stainless steel sheathed section of the unit may be coiled to a diameter not less than 50 cms.

24.4 The unit shall be packed in a rigid protective container in such a manner to avoid damage during transit.

* Non-corrosive non-toxic concentrate obtainable from Medical Pharmaceutical Developments LTD, Ellen Street, Portslade, Brighton BN 41 EQ, England.

25. IDENTIFICATION LABEL.

25.1 Each unit shall have a metal or plastic identification tag attached to it by plastic coated wire in such a way that damage to or contamination of the sheath is avoided.

25.2 The tag shall be approximately 100 mm by 30 mm and digits and lettering shall not be smaller than 1.5 mm. high.

25.3 The tag shall have the following information marked on it.-

The manufacturers identity of the thermocouple unit which must be unique.
Nominal composition of conductors, insulant, and sheath,
Customers order or contract identity.

A typical tag is illustrated below:-

Homiensa - C 1478
2-W5Re-Mo-13.0-BeO/47cm//0
2-405/426-Ac-10-MgO/348cm.
Dossier-Q/1977/10

Order or contract number originating from the ordering establishment

Code number of thermocouple cable as Para.2.

Manufacturers identification of the thermocouple unit.

26. TEST CERTIFICATE.

26.1 The manufacturer shall supply a test certificate for each thermocouple unit. The following data shall be recorded on the certificate:

(a) Manufacturer

(b) Serial number of thermocouple unit

(c) Date of completion of tests

(d) Conductor materials
(e) Sheath materials
(f) Insulant materials
— thermocouple and compensating cable.

(g) Loop resistance

(h) Cold insulation resistance

(i) Hot insulation resistance

(j) Calibration data

(k) Intermediate junction calibration data

(l) Radiograph serial number

(m) Statement of compliance with this specification. (including any relaxation (s) made with the approval of the customer). To be signed by responsible member of the firm which manufacturers the unit.

(n) Order number

27. INSPECTION AT MANUFACTURERS WORKS.

Thermocouple manufacturers inspection services have in the past been found somewhat inadequate in scope and execution, and it is felt that this mediocrity arises from insufficient appreciation of the special conditions which are pertinent to irradiation experiments. To secure an improvement in this important area, it is recommended that certain of the operations and tests detailed in this specification are witnessed or conducted by members of the organisation which orders the thermocouples. The inspectors should obviously be conversant with thermocouples generally, and be experienced in the inspection of small components and interpretation of radiographs. He should additionally have considerable experience in the specialised field of nuclear experiment construction.
To assist in this crucial function of irradiation experiment thermocouple inspection, the following notes are provided for guidance.

The standards of inspection are classified under three categories which are

<u>CATEGORY 1</u> The inspector should fully inspect all materials certificates, processes, and packing to ensure that all the clauses and conditions of the specification are adhered to without exception.

<u>CATEGORY 2</u> Process inspection is delegated to the manufacturing organisation, but the buying organisation inspector should visit the manufacturer to check storage and clean area conditions before manufacture commences and also to authenticate material certificates and materials.
Random visits should also be made to ensure that the conditions of the specification are being applied.

<u>CATEGORY 3</u> Inspection is limited to examination of documents supplied by or to the manufacturing organisation.

Inspectionby the buying organisation's inspector, shall not relieve the manufacturer of his responsibility to supply the ordered thermocouples in accordance with the specification.

The manufacturer shall:

(i) Contact the buying organisation before manufacture commences, to make the necessary identification of materials with makers certificates, to inspect the storage and assembly areas, and to determine the stages at which process inspection is required.

(ii) Arrange for the buying organisations inspector to have free access at all times, whilst work on the thermocouples is being performed, to all parts of the works which concern the order.

(iii) Keep the buying organisation inspector informed of work progress, and to notify him in advance when any processes or tests are to be carried out.

APPENDIX 1

The following solutions are recommended to obtain a solution which has a surface tension of less than 35 dynes cm^{-1}

3.5 mL of Teepol *
3.5 mL of octyl alcohol (octanol)
1 litre distilled water

Alternatively:

20 mL (by volume) of DECON 90 **
3.5mL of octyl alcohol (octanol)
1 litre distilled water

The surface tension can be simply checked by placing a capillary tube of about 1.0 mm bore on the surface of the water. The height to which the water rises is measured in cm. and the surface tension given by:
S.T. = 245.3 dh, Dynes cm^{-1} where d and h in cm.

* Trade name of Shell Chemical Co. Ltd., Shell Centre, London S.E.I. England

** Trade name of Medical and Pharmaceutical Developments Ltd. Ellen Street, Portslade, Brighton, Sussex, England.

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C.E.N., Grenoble, France
C.E.N., Mol, Belgium
C.E.N., Saclay, France
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G.F.K., Karlsruhe, B.R.D.
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B. Bufalari, Irradiation Services, J.R.C., Petten
R. Gottschlich , one time Irradiation Services, J.R.C. Petten presently Metallschlauch-Fabrik, Pforzheim
J. Stiff, Research Reactors Division, AERE, Harwell, whilst attached to J.R.C. Petten.

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Alfred Nobel

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